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13. ABSTRACT (Maximum 200 words)

Investigations of magnetic resonance of defects in epitaxial semiconductors resulted in a pioneering study of the defects present in thin film GaAs grown by MBE at temperatures between 190 and 300°C. These novel epitaxial semiconductor thin films are of great technological interest, e.g., as buffer layers to avoid sidegating of FETs or in ultrafast detectors with response times in the femtosecond range. A comprehensive analysis by magnetic resonance, infrared absorption, Hall effect, x-ray diffraction, and particle-induced X-ray emission showed that the transport in these very As-rich layers is dominated by a hitherto unknown kind of hopping conduction between localized arsenic antisite defects present in concentrations up to  $10^{20}/\text{cm}^3$  that are partly compensated by up to  $10^{18}/\text{cm}^3$  acceptors. The total concentration of excess As reached values of  $6 \times 10^{20}/\text{cm}^3$ , corresponding to  $[\text{As}]/[\text{Ga}] = 1.03$ . This was found together with a lattice expansion of up to 0.15%. Thermal annealing to temperatures higher than 500°C resulted in disappearance of the lattice expansion, a reduction of the antisite defect concentration by at least two orders of magnitude, and the disappearance of hopping conduction. A new superconducting phase with a transition temperature of 10K was discovered after In diffusion into GaAs.

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## I. INTRODUCTION.

The last years have opened up new avenues to miniaturization and higher integration of microwave and optoelectronic devices by hetero-epitaxial growth of semiconductor films on substrates of similar crystal structure but of different lattice parameters. At the same time, new problems for scientific research have been created, such as understanding and identification of defects due to mismatched lattices and the search for ways of diminishing these defects.

In 1988, this group at the University of California at Berkeley took up studies of different epitaxial structures, such as GaAs/Si, Ge/Si, and GaAs/GaAs in an attempt to investigate the characteristic defects generated at interfaces and within the epitaxial layers. The work was done within the project "Magnetic Resonance of Defects in Heteroepitaxial Semiconductor Structures" funded since April 15, 1988. During the first year of the contract, the unexpected emergence of novel GaAs layers grown by molecular beam epitaxy (MBE) at very low temperatures (LT), between 190 and 300°C, led us to concentrate the main effort in this project on the characterization of the defects in such layers.

The big interest in LT MBE GaAs started with the recognition of its beneficial role as buffer layer for III-V devices grown on SI GaAs.<sup>1</sup> It has been shown that GaAs MESFET performance can be substantially improved by using LT MBE GaAs buffer layers. In addition, LT-GaAs layers have been successfully used as gate isolation contacts in MISFET structures. More recently, photodetector response times in the sub-picosecond range open up new applications of such layers as active parts of devices.<sup>2</sup>

LT MBE layers are grown under As-rich conditions at substrate temperatures between 190 and 300°C, which is substantially lower than for normal GaAs MBE growth at 550 to 600°C. LT MBE GaAs layers are usually annealed at around 600°C in GaAs devices since the active device structure is grown on the top of the LT-GaAs at this temperature. The resulting annealed LT layer is highly resistive, providing even better isolation of GaAs devices than semi-insulating bulk GaAs.

Our studies resulted in the identification of arsenic antisite defects in LT MBE GaAs layers by electron paramagnetic resonance (EPR).<sup>3,4</sup> It was also found by means of transmission electron microscopy (TEM) that in spite of the low growth temperature, the LT GaAs layers were still crystalline. On the other hand, a considerable amount of excess arsenic (~1at. % for layers grown at 200°C), much higher than in any other kind of GaAs, was reported based on the results of particle-induced x-ray emission (PIXE) and analytical electron microscopy methods. Simultaneously, x-ray diffraction studies revealed a very substantial (~0.1%) increase in the lattice parameter compared to bulk liquid encapsulated Czochralski (LEC) GaAs.

In the following, the accomplishments of this project will be presented. The main objective was to determine defects present in LT MBE GaAs, their role in electron transport properties, the influence of annealing up to 600°C on the presence of defects and the change of conduction mechanism upon annealing.

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## II. MAJOR ACCOMPLISHMENTS OF THE CONTRACT

The research for this contract was concentrated on a pioneering, collaborative study of the defects present in as-grown thin film LT-GaAs grown by MBE at temperatures between 190 and 300°C and their change upon in-situ and ex-situ annealing. A comprehensive analysis by magnetic resonance, infrared absorption, Hall effect, x-ray diffraction, and particle-induced X-ray emission showed that the transport in these very As-rich layers in the as-grown state is dominated by hopping conduction between localized arsenic antisite defects present in concentrations up to  $10^{20}\text{cm}^{-3}$  and partly compensated by up to  $10^{18}\text{cm}^{-3}$  acceptors. The total concentration of excess As reached values of  $6 \times 10^{20}\text{cm}^{-3}$ , corresponding to  $[\text{As}]/[\text{Ga}] = 1.03$ . This was found to occur together with a lattice expansion of up to 0.15%. Thermal annealing to temperatures higher than 500°C resulted in disappearance of the lattice expansion, a reduction of the antisite defect concentration by at least two orders of magnitude, and the disappearance of hopping conduction. Optically detected magnetic resonance (ODMR) experiments using luminescence emission were successfully implemented, but the luminescence emission of low-temperature grown GaAs turned out to be too small for detection by ODMR.

### II.1. Off-stoichiometry of LT layers

Particle induced x-ray emission (PIXE) was applied to study the stoichiometry of LT layers.<sup>4,5</sup> Systematic measurements of layers grown at different temperature showed strong dependence of excess arsenic on growth temperature. LT GaAs grown at 190°C showed the highest deviation from stoichiometry, with the arsenic-to-gallium ratio ( $N_{\text{As}}/N_{\text{Ga}}$ ) varying between 1.026 to 1.030. This corresponded to the range of excess arsenic amount  $\Delta\text{As}$  ( $\Delta\text{As} = (N_{\text{As}} - N_{\text{Ga}}) / (N_{\text{As}} + N_{\text{Ga}})$ ) of 1.3 to 1.5 %. LT GaAs grown at 200°C was also highly nonstoichiometric with a  $N_{\text{As}}/N_{\text{Ga}}$  ratio of 1.016 to 1.020, corresponding to  $\Delta\text{As}$  between 0.8 to 1%. Differences in the stoichiometry of layers grown at nominally the same temperature were due to small variations in substrate temperature from run to run. LT GaAs layers grown at temperatures greater than or equal to 300°C were stoichiometric, within the sensitivity limit of PIXE. PIXE is sensitive to deviations from stoichiometry of  $\geq 0.1$  %.

The changes of  $\Delta\text{As}$  in LT GaAs layers were also traced versus annealing temperature. The stoichiometry of LT GaAs layers grown at 200°C showed no change with *in situ* annealing for temperatures as high as 600°C. However, LT GaAs layers annealed in a reducing atmosphere showed a monotonic decrease in  $\Delta\text{As}$  as the annealing temperature was increased. The LT GaAs epilayer began to lose As during the anneal at 300°C, and for anneals at temperatures greater than or equal to  $\sim 450^\circ\text{C}$ , the LT GaAs epilayer was stoichiometric, within the sensitivity of the PIXE technique.<sup>5</sup>

## II.2. Lattice parameter change of LT layers

X-ray diffraction measurements were made on both as-grown and annealed LT GaAs. The lattice parameter of as-grown LT GaAs increased monotonically as the growth temperature was reduced. For growth temperatures greater than 300°C, the LT GaAs lattice parameter was equal to the lattice parameter of SI GaAs substrate  $a_0 = 5.653 \pm 0.001 \text{ \AA}$  within experimental resolution. For LT GaAs epilayers grown at 260 and 220°C, the lattice parameters were 5.658 and 5.654 Å, respectively. For a number of LT GaAs epilayers grown at nominally 200°C, the lattice parameter varied between 5.568 and 5.660 Å. Similarly, for several LT GaAs layers grown at nominally 190°C, the lattice parameter varied between 5.560 and 5.561 Å. As in the case of stoichiometry studies, the slight variation in the lattice parameter for different LT GaAs layers grown at nominally the same temperature was due to small variations in substrate temperature from run to run.

Changes of lattice parameter in LT GaAs layers were also traced versus annealing temperature. Annealing of LT GaAs up to 600°C both in reducing atmosphere and *in situ* under an As overpressure resulted in monotonic decrease of  $\Delta a$ , where  $\Delta a = a_1 - a_0$  and  $a_1$  is the lattice parameter of LT layer and  $a_0$  of SI GaAs substrate. The decrease of LT GaAs layer lattice parameter was observed starting from an annealing temperature of 300°C, and for layers annealed at 450°C the lattice parameter was equal to that of the bulk GaAs within the experimental error.

## II.3 Optical absorption and magnetic resonance of arsenic antisite defects in LT layers

Electron paramagnetic resonance (EPR) of as-grown LT-GaAs resulted in the first identification of arsenic antisite defects in MBE-grown GaAs.<sup>3</sup> The intensity of the characteristic four-line quadruplet of  $\text{As}_{\text{Ga}}^+$  corresponded to up to  $5 \times 10^{18} \text{ cm}^{-3}$ , which suggested that compensating acceptors are present in that concentration, as well as *at least* this number of arsenic antisite defects. Optical absorption spectroscopy applied to LT MBE GaAs layers revealed the presence of a near-infrared absorption band characteristic of EL2 defects in the neutral charge state.<sup>5</sup> EL2 defects in bulk GaAs are commonly related to arsenic antisite defects. From the value of the absorption coefficient it was possible to determine the concentration of  $\text{As}_{\text{Ga}}$  defects in the neutral charge state  $\text{As}_{\text{Ga}}^0$  in LT layers. For layers grown at 200°C, the concentration was about  $10^{20} \text{ cm}^{-3}$ . It corresponds within a few percent accuracy to the total concentration of  $\text{As}_{\text{Ga}}$  since the amount of paramagnetic  $\text{As}_{\text{Ga}}^+$  defects as measured by EPR did not exceed  $5 \times 10^{18} \text{ cm}^{-3}$ .

The concentration of  $\text{As}_{\text{Ga}}^0$  defects in LT GaAs was a function of the growth temperature and the annealing treatment of the layer. It decreased with increasing growth temperature. For LT GaAs layers grown at 190 and 200°C, the  $\text{As}_{\text{Ga}}^0$  related defect concentration varied between  $1.5\text{--}2 \times 10^{20}$  and  $1.2\text{--}1.3 \times 10^{20} \text{ cm}^{-3}$ , respectively. A layer grown at 220°C showed  $9 \times 10^{19} \text{ cm}^{-3}$   $\text{As}_{\text{Ga}}^0$  related defects.

The effect of annealing on the concentration of  $\text{As}_{\text{Ga}}$  defects was determined by absorption measurements on LT GaAs grown at 200°C.<sup>5</sup> This annealing experiment was carried out in an atmosphere of forming gas. The concentration of  $\text{As}_{\text{Ga}}$  defects started to decrease for layers annealed at 300°C and was no longer measurable (detection limit about a few times  $10^{18} \text{ cm}^{-3}$ ) for layers annealed at 450°C. Excess As and lattice constant changed similarly, indicating that these three quantities are closely related. Investigation of layers annealed *in-situ* in the MBE machine yielded similar results concerning lattice expansion and antisite defect concentration, but no loss of excess As was observed.

Illumination of LT layers with white light at helium temperatures led to the *partial* quenching of the near infrared absorption in contrast to the well known *total* quenching of the EL2 absorption in bulk GaAs. The recovery of the absorption spectrum occurred at about 120K, the typical recovery temperature for semi-insulating GaAs.<sup>5</sup>

#### II.4. Ion-channeling studies of defects in LT GaAs

LT GaAs layers were also investigated using Rutherford backscattering (RBS) with PIXE, channeling studies along  $\langle 100 \rangle$ ,  $\langle 110 \rangle$  and  $\langle 111 \rangle$  directions. For all three channels, the yield of the backscattered ions when the beam is exactly aligned with a channel was significantly higher for the LT GaAs layer than for the SI GaAs standard, and the width of the As  $K_{\beta}$  channeling dip was significantly reduced. This indicated that the channels were obstructed by a high concentration of As-related defects that dechanneled the incident ion beam, corresponding to the high concentration of excess arsenic found by PIXE studies. This As does not appear to be in sites which are incommensurate with respect to the host, e.g., in precipitates, as this would produce much broader channeling distributions. Thus the origin of the dechanneling could be arsenic interstitials. These interstitials cannot be in the undistorted interstitial sites, as scattering by such interstitial defects should only be seen for the beam orientation along  $\langle 110 \rangle$  or  $\langle 100 \rangle$  channels for interstitials in tetrahedral and hexagonal sites, respectively. A possible model would be split interstitials, i.e., interstitials sharing a lattice site with a host atom, but more work is required to solve the question of the dominant lattice site of the excess As.

#### II.5. Transport properties of LT GaAs layers

Based on the temperature dependence of conductivity and the Fermi level position estimated from optical absorption and magnetic resonance measurements, it was concluded that, in as-grown layers, hopping conductivity within a band of arsenic antisite defects took place. The activation energy of hopping conductivity was found to be about 0.2 eV for samples grown at about 200°C. The data of transport measurements indicated the decrease in activation energy of conductivity from about 0.2 eV for the as grown layer to about 0.05 eV for the layer annealed at 450°C and higher temperatures.<sup>6,7</sup> Upon annealing, another thermally activated mechanism of conductivity gradually appeared. This mechanism, having an activation energy of 0.79 eV, appeared first at higher measurement temperatures and was easily distinguished for samples annealed at 450°C and higher annealing temperatures. It dominated almost the whole measured conductivity for the sample annealed at 600°C.

To explain the change in conductivity behavior after annealing for LT GaAs layers, the decrease in concentration of arsenic antisite defects was considered. When the amount of arsenic antisite defects decreased with annealing, another mechanism of conductivity besides hopping appeared, namely by means of thermally excited free carriers. The activation energy of this mechanism, 0.79 eV, was the same as in SI bulk GaAs. The conductivity by means of free carriers in conduction and valence bands dominated measurements at most temperatures in LT layer annealed at 600°C. Since the conductivity measurements were performed on samples consisting of LT layer on SI GaAs substrate, it was impossible to distinguish LT layer conductivity from substrate conductivity when the layer started to lose antisite defects and, simultaneously, hopping conduction in the arsenic antisite defect band. However, the following general conclusions on the conduction mechanism of LT layers could be drawn from the annealing experiments:

For as-grown LT GaAs layers, hopping conductivity within arsenic antisite defect bands dominated, conduction by free carriers in conduction and valence bands appeared upon layer annealing, and for layers annealed at 600°C hopping conduction was only observable well below room temperature, whereas at higher temperatures conduction by free carriers dominated. For this annealing temperature the conductivity value was lower than in the SI bulk GaAs substrate because of a low mobility due to a still-high defect concentration. The optical absorption studies showed that arsenic antisite defect concentration decreased with annealing, to below the detection limit in the case of 600°C annealed layers, but this could mean a residual concentration as high as  $10^{18} \text{ cm}^{-3}$ .<sup>8</sup> An alternative explanation for the good device isolation properties of annealed LT-GaAs has been proposed by Warren, Woodall et al.<sup>9</sup>: Formation of metallic As-precipitates<sup>10</sup> might result in complete depletion of the LT GaAs through overlap of individual spheres of depleted regions around each precipitate; however, it is not yet clear whether all annealed LT GaAs layers showing the beneficial device isolation effects indeed contain enough As precipitates to account for such depletion.

## **II.6. Optically Detected Magnetic Resonance Experiments**

An experimental set-up for the investigation of optically detected magnetic resonance was completed, utilizing the existing He gas flow cryostat. This equipment allows us to detect changes in the photo-luminescence emission of a thin film sample due to paramagnetic resonance transitions of the electron in the excited (=starting) or ground (=final) state. A first experiment with GaP, yielding the triplet ( $S=1$ ) ODMR signal of the  $P_{\text{Ga}}^+$  antisite defect<sup>11</sup> was successful. All attempts to investigate any ODMR signal from as-grown LT GaAs layers failed due to the lack of sufficient light emission caused by the high defect concentration in these layers.

## **II.7. Superconductivity of GaAs**

In the final months of this contract, evidence for superconductivity in GaAs with a transition temperature of 10K was detected in magnetic resonance experiments with LT-GaAs samples.<sup>12</sup> This observation was confirmed by conventional susceptibility measurements with a

SQUID magnetometer. A patent on this invention was filed by the University of California. Since then, it has been shown that indium plays a role in the superconducting phase.<sup>13,14</sup> It was found that In that was used for the thermal contact between substrate and sample holder in the MBE machine diffuses even at temperatures below 600°C from the wafer backside into the sample.

### III. Future Work

For the continuation of the research described in this report, a proposal has been submitted to AFOSR with the title "Electronic properties of low-temperature grown III/V thin films". This proposal suggests focusing in the coming years on *annealed* LT-GaAs thin films which are the structures of practical interest. It is planned to study the defects in annealed LT-GaAs through transport measurements and new types of optical absorption and magnetic measurements in conjunction with structural studies of the same layers. In addition, the ability to obtain hitherto unavailable defect concentrations, e.g., of As<sub>Ga</sub> antisite defects in LT-GaAs, allow new kinds of defect characterization, e.g., by studying the influence of light on the lattice constant of LT-GaAs layers. It is further proposed to extend this work to other LT-grown III/V compounds and alloys.

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## VI. PUBLICATIONS.

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9. Influence of Growth Rate and Temperature on the Structure of Low-Temperature GaAs, Z. Liliental-Weber, A. Claverie, P. Werner, W. Schaff, and E. R. Weber, in: *Defects in Semiconductors 16* (Ed. G. Davis, G. deLeo, and M. Stavola), Materials Science Forum vol. 83-87, (Trans Tech Zürich 1992), p. 1045.
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## **VII. PRESENTATIONS AT CONFERENCES.**

1. 16th annual PCSI Conference, Physics and Chemistry of Semiconductor Interfaces, Bozeman, Montana, February 7-9, 1989.
2. WOCSEMAD 1989, Hilton Head, February 20-22, 1989.
3. Workshop on Instabilities in III-V Devices, Sedona, Arizona, April 24-26, 1989.
4. Gordon Research Conference, Point and Line Defects in Semiconductors, Plymouth State College, Plymouth, New Hampshire, July, 1989
5. Spring Meeting of the Materials Research Society, San Francisco, California, April 16-21, 1990.
6. Workshop on Low Temperature GaAs, San Francisco, California, April 21, 1990.
7. 6th International Conference on Semi-Insulating III-V Materials, Toronto (Canada) 1990, May 13-16, 1990.
8. 20th International Conference on the Physics of Semiconductors (ICPS-20), Thessaloniki (Greece), August 6-10, 1990.
9. Fall Meeting of the Materials Research Society, Boston, Massachusetts, November 26 - December 1, 1990.
10. 16th International Conference on Defects in Semiconductors (ICDS-16), Bethlehem, Pennsylvania, July 22-26, 1991.
11. Fall Meeting of the Materials Research Society, Boston, Massachusetts, December 2-6, 1991.

## **VIII. Personnel**

Dr. Maria Kaminska, (Ph.D. 1979), visiting scientist,

Thomas George, graduate student (Ph.D. 5/1990),

Waifan Yau, graduate student (Ph.D. 12/91),

Rosa Leon, graduate student, Ph.D. candidate (Ph.D. expected 7/92).